Standard Operating Procedure for the Analysis of Particulate-Phase Organic Carbon

LG207

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1.0 SCOPE AND APPLICATION

1.1 This method describes the analysis of glass fiber filters from particulate-phase Great Lakes water samples for particulate organic carbon (POC) using an elemental analyzer and a computer data system. This SOP covers standard and instrument preparation, instrument calibration and maintenance, elemental analysis of carbon, and calculation of results.

1.2 The working range of analysis is 0.0 - 100.0 µg carbon.

2.0 SUMMARY

2.1 Sub-samples of the exposed glass fiber filters are placed into small tin capsules to be placed on the autosampler of a Carlo Erba Elemental Analyzer 1108. The autosampler tin capsules are dropped a vertical quartz reactor tube inside a 1,000°C furnace. The carrier gas temporarily enriched with oxygen oxidizes the sample. Quantitative oxidation is achieved when the resulting mixture of gases pass over a tungsten anhydride catalyst. The gas mixture then passes over the elemental copper where excess oxygen is removed and nitrous oxide is reduced to elemental nitrogen. The sample gases then pass through a packed chromatographic column to be separated, eluted, and detected by a thermal conductivity detector (TCD). Organic carbon is quantified by the external standard method.

2.2 The elemental analyzer comprises a combustion furnace, gas chromatographic oven, and a thermal conductivity detector. This instrument can be configured to detect carbon, hydrogen, nitrogen, and sulfur simultaneously. The instrument can also be configured to detect oxygen through the use of a separate determination mode. The analyzer is equipped with a pneumatic autosampler and a PC based computer data generation system. This instrument uses one of two available furnaces to house a catalytic reactor tube. The reactor is packed with a tungsten anhydride oxidation catalyst in its upper chamber and a copper elemental reduction reactor in its lower chamber. Both chambers are packed into the chromatographic column. Sample components are separated into elemental species which are detected by the thermal conductivity detector (TCD).

3.0 SAMPLE HANDLING

3.1 Samples are filtered by vacuum through 47-mm glass fiber filters with a 0.7-µm pore size. The filters retaining particles are collected into clean aluminum foil envelopes and frozen at -10°C until analysis.

4.0 INTERFERENCES

4.1 Improperly handled or damaged filters should be discarded.

4.2 When using aluminum foil for ashing and storage, be sure that the shining side faces out and the dull side faces the sample filters or container mouths.

4.3 Failure to install properly a water trap outside the detector of the elemental analyzer will result in an unstable baseline. Be sure that the anhydrone reagent used is fresh and that the quartz wool is clean.

4.4 An exhausted or incorrectly packed reactor tube can result in elevated concentrations of nitrogen or an irregular baseline.

5.0 APPARATUS AND SUPPLIES

5.1 Tin sample containers (Fisons 24006400)

5.2 Reactor tubes (Fisons 46820000)

5.3 Anhydrone (Fisons 338219000)

5.4 Quartz wool (Fisons 33822200)

5.5 Copper wire pieces (Fisons 33835310)

5.6 Viton O-ring for water trap (Fisons 29013603)

5.7 Viton O-ring for reactor tube (Fisons 29032910)

5.8 Tungstenic anhydride (Fisons 33835420)

5.9 EDTA disodium salt (J.T.Baker 8993-01)

5.10 Potassium hydrogen phthalate (Fisher P243-100)

5.11 Concentrated sulfuric acid (Fisher A510-500)

5.12 47-mm glass fiber filters with 0.7-µm pore size (Whatman 182547)

5.13 50-mL micro pipettor (Daigger Scientific G20537F)

5.14 OMI-I indicator tube (Supelco 2-3900)

5.15 50 x 9 mm plastic Petri dishes

5.16 400-mL beakers

5.17 12-mm punch (bor#6), 6-mm punch (bor#3)

5.18 Stainless steel forceps

5.19 Class A volumetric flasks

5.20 Class A volumetric pipets

5.21 Concentrated hydrochloric acid (Fisher A466-500)

5.22 Pesticide-residue grade acetone (J.T.Baker Photrex 9010-03)

5.23 Carlo Erba EA 1108 Elemental Analyzer with pneumatic autosampler

5.24 Carlo Erba Eaber 200 PC-based data system

5.25 Specialty compressed gases

5.25.1 Helium, 99.999% UHP grade

5.25.2 Oxygen, 99.999% UHP grade

5.25.3 Air, for pneumatic valve operations of the instrument and autosampler

6.0 REAGENTS AND STANDARDS

6.1 Reagents, standards, and controls should be stored in appropriate containers with well-fitting lids. All reagent container labels should contain the following information:

Information	**Example**
Identity:	0.2 N Hydrochloric Acid
Date: (mm/dd/yy)	04/06/97
Initials of Prep Analyst	I.M.F.

In addition to identity, date, and preparation data, standard and control solution storage containers must state the concentration of the solution contained as follows:

Concentration: 2000 mg C/L

Reagent, standard, and control containers can also contain other appropriate information, such as optimum storage conditions or analytical designation. **All reagents, standards, and controls should be prepared using carbon-free reagent water.**

<u>Be sure to use only Class A volumetric flasks and volumetric pipets when preparing all reagents, standards, and controls</u>.

6.2 Reagents

6.2.1 **Reagent water**: Distilled and deionized carbon filtered ultra pure water.

6.2.2 **0.2 N Hydrochloric Acid**: In a 1-L volumetric flask, mix 500 mL organic-free water with 16.6 mL concentrated hydrochloric acid. Dilute to volume with reagent water. This reagent can be stored in a plastic container at room temperature indefinitely or until exhausted.

6.3 Standards

6.3.1 **Stock Calibration Standard, 2000 mg C/L**: Dry 6 grams potassium hydrogen phthalate ($KHC_8H_4O_4$) at 110°C for two hours. Allow to cool in a desiccator. In a 1-L volumetric flask, dissolve 4.2509 grams $KHC_8H_4O_4$ in 500 mL reagent water. Add 0.2 mL concentrated sulfuric acid (H_2SO_4). Dilute to 1 L with reagent water. Store in a clean glass bottle at 4°C. Prepare this stock standard fresh monthly.

6.3.2 **Working Calibration Standards, 5 - 100 mg carbon/ 50 µL spike**: The following carbon working standards for the analytical range are prepared by dilution of intermediate stock standard 6.3.1 as follows:

mL Stock 6.3.1 diluted to 100 mL	µg carbon/50 µL spike
10.0	10.0
25.0	25.0
50.0	50.0
75.0	75.0
100.0	100.0

6.3.3 Working calibration standards should be prepared fresh monthly. Store these standards in clean glass bottles at 4°C.

6.4 Control Standards

6.4.1 **Stock Control Standard, 2000 mg carbon/L**: Dry 10 g ethylenedinitrilo-tetra-acetic acid, disodium salt, dihydrate (EDTA, disodium salt, $C_{10}H_{14}N_2Na_2O_8 . 2H_2O$) at 70°C for one hour. Allow to cool in a desiccator. In a clean 1-L volumetric flask, dissolve 6.1983 g $C_{10}H_{14}N_2Na_2O_8 \bullet 2H_2O$ in 500 mL organic-free water. Add 0.2 mL concentrated sulfuric acid. Mix well and slowly dilute to 1 L with reagent water. Store in a clean glass bottle at 4°C. Prepare fresh monthly.

6.4.2 **Working Control Standards**: The following control standard solutions are prepared by dilution of the control standard stock 6.4.1 as follows:

mL Stock EDTA diluted	µg carbon/50 µL spike
15.0 mL → 100 mL	15.0
20.0 mL → 25 mL	80.0

6.4.3 Working control standards should be prepared fresh monthly. Store in clean glass bottles at 4°C.

7.0 PROCEDURE

7.1 Preparation of the Elemental Analyzer

7.1.1 Refer to the elemental analyzer and PC data system operating manual for directions for routine operations. **If the instrument is being operated on board a research vessel, be sure to isolate the instrument from sources of vibration and mount the instrument securely.**

7.1.2 Locate the main power source switch on the rear panel of the instrument. The instrument power switch can remain in the off position when not in use for analysis.

7.1.3 The instrument has three functional systems which require compressed gases for operation.

7.1.3.1 Helium is used as the carrier gas. The helium gas line should contain a oxygen-hydrocarbon trap for purity maintenance. The helium used should be 99.999% ultra high purity grade gas. The supply pressure should be set in a range of 200-300 kPa at the tank.

7.1.3.2 Oxygen is used for combustion in the reactor tube. The oxygen should be 99.999% ultra high purity grade gas. The supply pressure should be set to 100 kPa at the tank.

7.1.3.3 Compressed air is used to operate pneumatic valves in the instrument and to activate the autosampler. Set the supply pressure in a range of 350 - 400 kPa at the tank.

7.1.4 The instrument's analytical configuration is determined by the CHNS (carbon-hydrogen-nitrogen-sulfur) packing contained in the reactor tube. To pack the reactor tube, mark the heights of the packing layers on the sides of the quartz tube with a permanent marker. Fill each layer with the appropriate material to its specific mark. The materials are held in place with quartz wool. A Viton o-ring should be placed flat side down on the empty end of the packed quartz reactor tube.

7.1.5 Lower the reactor tube into the left furnace chamber copper end first so that the copper end is on the bottom. Adjust the inside o-ring so that it is about an inch from the top of the reactor tube. Attach the auto-sampler to the top of the reactor tube. Place a nut and washer followed by an o-ring flat side up on the coupling to be attached to bottom of the reactor tube.

7.1.6 The water trap on the detector line is maintained by filling the glass tube with anhydrone (magnesium perchlorate). Use 5 mm quartz wool to hold the material in place. Connect the detector line fittings to the water trap and clip it into the mounting bracket in front of the chromatographic oven. **Check the water trap daily. Repack the trap when needed.**

7.1.7 **Perform a leak check daily before the first analysis.** Gas seepage at joints along the gas lines can be detected with the use of a liquid leak detector. To determine possible leaks within the analyzer, turn on the power switch and depress the filament stand-by button located on the left corner of the instrument's control panel. Set the air pressure to 350 kPa using the pressure regulator on the front panel of the instrument. Wait 2 minutes. Turn off the air tank pressure regulator. There is no gas seepage at the joints if a constant 350 kPa pressure is maintained for 2 minutes. Then remove caps from the helium and oxygen measuring and reference vents. Turn the pressure regulators to zero. The regulator control knobs are located on the lower front control panel. Adjust the pressure regulator to 100 kPa for helium, and to 150 kPa for oxygen. Wait 2 minutes. Turn off the front panel valves for helium and oxygen. There is no gas seepage from the analyzer if the respective set pressures are maintained. Replace the caps with the proper fittings. Release the filament stand-by button.

7.1.8 Set the **helium analytical flow rate to 100 mL/min** using the front panel pressure regulator. Adjust the flow rate using a soap-bubble flowmeter at Vent M on the lower front instrument panel. Set the **helium reference flow rate to 40 mL/min** using the front panel regulator. Adjust the flow rate using a soap-bubble flow meter at the PURGE Vent on the lower front instrument panel. Set the **oxygen flow rate to 12 mL/min** using the front panel pressure regulator. Adjust the flow rate using a soap-bubble flowmeter at the OXYGEN Vent on the lower front instrument panel.

7.1.9 Set the instrument control panel settings as follows:

7.1.9.1 **Left furnace**, 1000°C

7.1.9.2 **Right furnace**, 500°C

7.1.9.3 **Oven temperature**, 70°C

7.1.9.4 **Filament temperature**, 180°C

7.1.9.5 **Cycle** (the time needed to analyze 1 sample), 180 seconds

7.1.9.6 **Sample start** (seconds elapsed into the cycle when the sample is dropped into the reactor tube), 15 seconds

7.1.9.7 **Sample stop** (seconds elapsed into the cycle when the autosampler advances to the next sample), 40 seconds

7.1.9.8 **Oxygen inject** (seconds elapsed into the cycle when oxygen enrichment ceases), 35 seconds

7.1.9.9 **Peak enable** (seconds elapsed into the cycle when data acquisition begins), 0 seconds

7.1.10 Settings for Sample Start and Sample Stop may require adjustment to optimize complete sample combustion during the oxygen inject interval. Sample combustion can be viewed through the *view finder* located on the face of the autosampler. It is recommended that the operator view sample combustion to confirm optimal control settings. In the case of inadequate sample combustion, increase the Oxygen Inject period. **Always set the <u>Sample Stop</u> period 5 seconds greater than the <u>Oxygen Inject</u> period.**

7.2 Instrument Start-Up

7.2.1 Flip the main power switch to 'ON' position.

7.2.2 Turn the compressed gases on at the tanks. Then turn the instrument purge and carrier valves to the 'ON' position from the controls on the front panel of the instrument. Release the filament and furnace stand-by by depressing the respective controls on the front panel of the instrument. Allow the furnace to achieve the 1000°C set point.

7.2.3 **Prior to every analysis always perform a leak check before adjusting gas flow rates.**

7.2.4 Activate the data system. Prompt the data acquisition software.

7.3 Instrument Calibration

7.3.1 Using a clean 12-mm diameter boring tool, bore out 6 filter discs from the ash furnace.

7.3.2 Using the micro-pipettor, spike each bored disc with 50 µL of calibration standard. **Rinse the pipettor five times with reagent water. Then initialize the pipettor by rinsing the tip five times with the standard. Between standards aliquot applications rinse the pipettor five times with organic free water.** Allow the discs to dry.

7.3.3 While the spiked filters are drying, input calibration information into the sample table of the data acquisition system. Start the analysis sequence with a by-pass, an empty sample container into which standards are added from the lowest to the highest concentration. Input the concentration of carbon spike the standard in mg in the 'sample weight' column of the data table. In the 'sample type' column of the data table, input "standard". In the 'standard type column' of the data table, input "POC". **Verify that the analysis sequence listed in the sample table is accurate. These instructions guide the data software in correct data processing. The first and last discs must be properly noted in the sample table.**

7.3.4 Using clean stainless steel forceps, fold the spiked discs into eighths and place them into individual tin sample containers. Pack the spiked filters completely inside of the tin containers. Load the calibration standards into the autosampler in the same order as the analysis sequence created in the sample table.

7.3.5 On the instrument's control menu, select the run command. Nitrogen (N_2) will elute first, followed by carbon as carbon dioxide (CO_2). Confirm that the retention time listed in the component data table of the data acquisition software is the same as the actual retention time. If the difference between actual and listed retention times is too large, the data system will not identify the peaks properly. Enter the correct retention times into the method table and repeat the calibration analysis. **The correlation coefficient for this five-level calibration plot should be 0.995 or better. If this criterium is not satisfied, the calibration analysis should be repeated.**

7.3.6 When the calibration criteria have been satisfied, use a 12-mm boring tool to bore out three 12-mm diameter discs from the muffled discs. Pipet 50 µL of the control standards onto two of the 12-mm discs and 50 µL of water onto the third one. **Before adding standards to the disc be sure to first rinse the pipettor five times with organic-free water and the standard solution.** Allow all three discs to dry.

7.3.7 While the control standard discs are drying, input the control and blank data into the sample table of the data acquisition system. In the 'sample type' column, enter unknown. In the 'sample weight' column, enter 100. Verify that the listed analysis sequence is correct, and that the first and last discs to be analyzed have been appropriately designated.

7.3.8 Load the control and blank sample discs into the autosampler and analyze. If the quality control sample values determined by analysis fall within the given tolerance ranges, proceed with the particulate organic carbon analysis of routine field samples. **In the event that any determined sample value falls outside the given tolerance range, repeat steps 7.3.6 and 7.3.7, the control and blank standard analysis. If the repeated set of controls render determined values that are outside the tolerance range, repeat steps 7.3.1 - 7.3.5 to recalibrate the instrument.**

7.4 Analysis of Routine Field Samples

7.4.1 Carefully open the aluminum foil envelope containing the folded sample filter. Take a small piece of the aluminum foil envelope and place it inside a 50 mm x 9 mm Petri dish, shiny side down.

7.4.2 While the sample filter is folded in half, use a clean 12-mm boring tool to cut two 12-mm sample discs, resulting in four discs. Be sure that an area of the 47 mm filter well covered with particles. Separate the four discs and place them particulate side up on the foil in the Petri dish. Cover the dish to protect the discs from contamination. Allow the discs to dry in the dish. **Each dish should be labeled with site and station information, date and time of sampling, and the initials of the analyst who performed the sampling routine.**

7.4.3 While the sample discs are drying, input sample information into the sample information table of the data acquisition system. In the 'sample type' column, enter "unknown". In the 'sample weight' column, enter "100". Verify that the information entered into the sample table is accurate and that the first and last discs to be analyzed have been properly denoted.

7.4.4 Use clean metal forceps to fold the dry discs into eighths and pack each folded disc completely into a separate sample container. Load the packed container into the autosampler in the same sequence listed in the sample information table of the data acquisition system. Select the 'run' command on the instrument operation panel. The data acquisition system will automatically generate sample data.

7.4.5 If the concentration of routine field sample higher than the calibration range, four additional discs should be prepared by using boring tool # 3 and analyzed as described previously.

7.5 Instrument Shut-Down

7.5.1 To shut the instrument down for a short period of time, depress the furnace and filament buttons on the instrument front control panel. Then shut off the oxygen and air supplies.

7.5.2 To shut the instrument down for the day, depress the furnace and filament buttons on the instrument front control panel. Shut off all three gas supplies at the respective tanks.

Remove the water trap at the analyzer's detector line and dispose of the waste properly. Remove and cap both ends of the chromatographic column and use an adaptor to connect the column inlet to the column outlet. Cap the three gas vents on the lower front panel of the instrument. Switch the main power switch to the 'off' position.

8.0 CALCULATIONS

8.1 The resulting mass of carbon from the analysis of four 12-mm routine field sample discs is calculated by multiplying the sum of the four determined carbon masses by an area correction factor (X) and dividing this product by the volume of aqueous sample filtered. To calculate the concentration of carbon on the particulate material coating the 47-mm filter multiply the sum of the carbon masses from each of the four 12-mm field sample discs by an area. The mass is reported in mg carbon/L.

$$mg/L = \frac{(sum\ of\ the\ determined\ masses\ of\ 4\ discs) \times X \times X_1}{Volume\ of\ sample\ filtered\ (L)}$$

8.2 Since the filtration apparatus exposes only 38 mm of the 47-mm filter surface, and, from this 38 mm surface, four 12-mm discs are used in carbon analysis, the correction factor (X) is a ratio calculated as follows:

$$\text{AREA}_{38} = 3.14159\ (19\ \text{mm})^2 = 1134\ \text{mm}^2$$

$$\text{AREA}_{12} = 3.14159\ (6.0325\ \text{mm})^2 = 114.33\ \text{mm}^2$$

$$\text{Correction Factor (X)} = [(\text{AREA}_{38}/4)\ (\text{AREA}_{12})] = 2.48$$

8.3 If discs was prepared by using boring tool # 3, the additional correctional factor (X_1) has to be added to the formula in 8.1.

$$\text{Correction Factor } (X_1) = [(\text{AREA}_{12})/(\text{AREA}_6)] = 2.827$$

9.0 QUALITY CONTROL

9.1 Refer to the Chapter 2 Introduction for definitions of quality control samples and information regarding quality control procedures, such as QC sample IDs and labeling.

9.2 The minimum acceptable correlation coefficient (r) is 0.995.

9.3 The following QC samples must be prepared and analyzed at the minimum frequency indicated.

QC Sample Type		Frequency	Acceptance Criteria
External	Field Reagent Blank (FRB)	One per basin[a]	≤ 5.0 µg/L or less than one-tenth associated field sample concentrations, whichever is greater
	Lab Duplicate (LD1)	One per basin[a]	RPD ≤ 20%
Internal	Calibration	At the beginning of each batch	r ≤ 0.995
	High Check Standard (CH)	At the beginning & end of each batch or 1 per 40 samples, whichever is more frequent	80.0 ± 16.0 µg/L
	Low Check Standard (CL)	At the beginning & end of each batch or 1 per 40 samples, whichever is more frequent	15.0 ± 3.0 µg/L
	Laboratory Reagent Blank (LRB)	At the beginning & end of each batch or 1 per 40 samples, whichever is more frequent	≤ 5.0 µg/L
	Method Detection Limit (MDL)	Once per year and each time a significant change is made to the SOP	5.0 µg/L

[a] A field duplicate, lab duplicate, and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

9.4 Assessment

9.4.1 The analyst must compare analytical results to the acceptance criteria listed in Section 9.3 to identify QC failures. If the results are outside the acceptance criteria, the analyst should first review their calculations for errors and if none are identified, they must follow the corrective action procedures listed in Section 9.5.

9.5 Corrective Actions

9.5.1 Corrective action procedures will often be handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and any other potential sources of error. If failure occurs and an error is identified, the analyst should re-run quality control and RFS samples in the entire analytical batch to confirm the results. Because external QC samples are collected and prepared during the survey and provided to the contractor or grantee laboratory, a single rerun to confirm results is sufficient when all other QC samples are within acceptance criteria. If the CH, CL or LRB does not meet the specified criteria, corrective action will take place and affected samples will be rerun. For analyses conducted onboard, if the problem persists or cannot be identified, the matter must be referred to the Chief Scientist for further investigation. Depending upon the Chief Scientist's evaluation, the analyst may or may not be required to prepare and re-run the samples again. Additionally, if the results are significantly different than the expected concentrations based on historical data or related samples, then the analyst may split the RFS sample in the laboratory and analyze the splits. Once a decision is made, full documentation of the corrective action procedures and assessment of the final result must be filed with the WQS QM Technical Lead (Marvin Palmer) or the GLNPO QM. For analyses conducted at contract or grantee laboratories, this information can be included with submitted data. When contractor or grantee laboratories have a question regarding acceptable corrective actions, they should contact the Biology Technical Lead or Limnology Technical Lead as appropriate for instruction at a time when corrective action can still be taken.

9.6 Data Reporting/Recording

9.6.1 When corrective actions are not feasible or do not resolve QC failure, the analyst is responsible for identifying all failed QC samples and RFS samples. If analyses are being conducted onboard, the analyst should document the QC information on the hard-copy Field Information Recording Forms (Appendix H). If analyses are being conducted by contract or grantee laboratories, the analyst should document all QC information with the submitted data.

10.0 SAFETY PRECAUTIONS AND WASTE DISPOSAL

10.1 Refer to the GLNPO Safety, Health, and Environmental Compliance Manual and the instrument procedural operations manual for specific details on applicable personal health and safety issues and instrumental, chemical, and waste handling procedures, and accident prevention.

10.2 All containers storing reagents, standards, controls, blanks, and wastes used in the lab must be properly identified through appropriate labeling and hazard definition.

11.0 PREVENTATIVE MAINTENANCE AND TROUBLE-SHOOTING

11.1 For routine maintenance, refer to the instrument procedural operations manual for manufacturer suggested methods and routines.

11.2 When the detector response for nitrogen consistently displays increased concentrations in consecutive chromatograms, the copper metal in the reactor tube has reached complete copper oxidation. Each reactor tube bearing an approximate 80.0 mm packing of copper can complete roughly 75 analytical runs from calibration through final control checks.

11.3 An unstable baseline could indicate the exhaustion of the tungsten anhydride in the reactor tube.

11.4 Refurbishing the reactor tube

11.4.1 Depress the filament stand-by button on the control panel.

11.4.2 Remove the coupling from the bottom of the reactor tube. Disconnect the autosampler from the top of the reactor tube.

11.4.3 Remove the reactor tube from the furnace. Remove the quartz wool at top and remove the tungstic anhydride. Remove the quartz wool plug and the copper metal from the bottom of the reactor tube.

11.4.4 Repack the top of reactor tube with 10 mm quartz wool before turning upside down and adding 80 mm tungstic anhydride. Pack the tungstic anhydride into place with 10 mm quartz wool before packing in 80 mm of fresh copper wire pieces. Use 10 mm quartz wool to hold the copper pieces in place. Turn the reactor tube tungstic anhydride end up.

11.4.5 Scrape out the dioxin residue from the top of the reactor tube. Replace the quartz wool, if needed.

11.4.6 Reconnect the top of the reactor tube to the autosampler, and connect the bottom to the coupling, using new Viton O-rings on the reactor tube. **Be sure that the flat sides of the O-rings face away from the reactor tube at both ends.**

11.4.7 Perform a leak check on the pneumatic systems.

11.5 Be sure that the water trap is functioning properly and is not spent. Water trapped in the detector, indicated by baseline problems, can impede carrier gas flow.

11.6 If the water trap is not spent, the reactor tube has been freshly repacked, and baseline interferences are still evident, check the gas supply lines and the chromatographic column for blockages. Refer to the instrument procedural operations manual for manufacturer suggested course of action.

11.7 **Perform a thorough leak check before every analysis.**

12.0 REFERENCES

12.1 GLNPO Organic Analysis Standard Operations Procedure, Revision 2.

12.2 Instrumental Procedural Operations Manual for the Carlo Erba EA 1108 Elemental Analyzer.

12.3 PC System Procedural Operations Manual for the Carlo Erba Eaber 200 Data Acquisition System.

12.4 GLNPO Safety, Health, and Environmental Compliance Manual.